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MONTHLY PROGRESS REPORTS

Period Covered

May 1 - May 31, 1973

CONFIDENTIAL

Date Issued: June 15, 1973

2022158631

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2022158632

CHARGE NUMBER: Various
PROGRAM TITLE: Analytical Investigations
WRITTEN BY: M. C. Bourllas, D. C. Watson, and E. T. Oakley
DATE OF REPORT: June 14, 1973

I. SMOKE CONDENSATE STUDIES (6908)

A limited study of the chemical composition of whole smoke condensate has been initiated at the request of Mr. W. F. Kuhn. Smoke condensate from a series of twelve cigarette types will be analyzed for total nitrogen, nicotine, phenols, and pH. Initial results on condensate from Kentucky Reference 1R1 cigarettes showed excellent precision. The RSD at the 2 σ level is $\pm 4\%$ for phenols, $\pm 5\%$ for total nitrogen, $\pm 2\%$ for nicotine and $\pm 11\%$ for pH (H^+ ion concentration).

II. SORBIC ACID IN RCB

The sorbic acid AutoAnalyzer channel has been interfaced with the computer in order to facilitate the analysis of the extremely heavy load of samples.

III. SULFIDES IN GAS PHASE

Work has begun on a colorimetric AutoAnalyzer method for the determination of sulfides in gas phase. The ultimate goal is to incorporate this analysis into the puff-by-puff gas phase analysis as a fifth channel.

IV. Methods were developed, in conjunction with Fabriques de Tabac Reunies SA and the CORESTA sub-committee, for the determinations of various pesticide residues in tobacco. These include γ -BHC, Heptachlor, Aldrin, Dieldrin, p,p'-DDE, o,p'- and p,p'-TDE, and o,p'- and p,p'-DDT. The pesticide residues cover the concentration range down to 0.02 ppm.

V. A number of the components of the gas phase of cigarette smoke were identified using GC/MS. Using this technique, the identities of several of the multi-component GC peaks were determined.

VI. Mainstream puff-by-puff TPM, nicotine and water delivery of Marlboro Monitor cigarettes has been measured near the char-line and compared to that obtained at the end of the rod. From this data, the amount of these components absorbed on the rod can be calculated.

J. Will

/nwp

2022158633

PROJECT NUMBER: 0105

PROJECT TITLE: PHYSICAL CHEMISTRY OF SMOKE AND FILTRATION

PERIOD COVERED: May 1 to May 31, 1973

PROJECT LEADER: H. A. Hartung

SORPTION IN FILTERS

Two experimental techniques are being utilized to characterize the interaction of organic smoke components and filter materials. One involves the fixed bed adsorption (FBA) of nicotine vapors on cellulose acetate (CA) and paper. A model adapted from chemical engineering theory has been tested with data from additional combinations of vapor concentration, filter materials and flow rates. The results were very good and they have shown that untreated CA has a greater capacity than paper for nicotine removal. (1) The second experimental program utilizes gas chromatography (GC) to measure vapor sorption parameters. Specific and non-specific chemical group effects are being derived with CA and a homologous series of compounds. The tests are also being extended to elevated temperatures to obtain heats of adsorption. (2)

BASELINE STUDIES AND MATHEMATICAL MODELLING (4,5)

Puff-by-puff deliveries of TPM, tar and nicotine were found to fit well to the general equation

$$\text{DELIV} = \alpha_1 \times \exp(b_2 \times \text{BUTT}) + \alpha_2 \times \exp(b_2 \times \text{BUTT})$$

Best fit values for the four coefficients were derived by a non-linear least squares regression technique. At relatively long distances from the coal only one term in this equation is important. It gave a filtration coefficient of 1% per millimeter for TPM, tar and nicotine. The sum $\alpha_1 + \alpha_2$ is an indication of amount generated on the puff at zero butt. The values obtained were

	<u>α_1</u>	<u>α_2</u>	<u>$\alpha_1 + \alpha_2$</u>
TPM	4.4	8.5	12.9
Tar	4.3	5.0	9.3
Nicotine	.37	.54	.91

ISOLATION OF FLAVOR CONSTITUENTS

The evolution of various gases and vapors from tobacco is being investigated during heating in an inert gas stream. A series of measurements on CO and CO₂ is being carried out with various tobacco types and blends. That program is nearing completion and the test method is being extended to other vapors. Considerable NH₃ evolution was detected at 280°C, and evidence for mercaptans was obtained.

2022158634

Page 2.

Work on the sparging process to isolate flavor constituents has been stymied by extensive delays in the construction of new apparatus (3).

/gdc

References:

- (1) Simpson, Notebook 6219, pp. 65-82.
- (2) Dwyer, Notebook 6308, pp. 1-20.
- (3) Osborne, Notebook 6143, pp. 27-30.
- (4) Stewart, Notebook 5559, p. 54.
- (5) Hartung, memo to D. A. Lowitz and A. C. Lilly, May 15, 1973.

HA Hartung

2022158635

CHARGE NUMBER : 0108
 PROGRAM TITLE : MECHANISM FOR SMOKE FORMATION
 PERIOD COVERED: May 1 - May 31, 1973
 PROJECT LEADER: W. R. Johnson
 DATE OF REPORT: June 7, 1973

I. OXIDES OF NITROGEN IN CIGARETTE SMOKE^{1,2}

A method was developed for the determination of nitrogen dioxide in sidestream smoke. The method was adapted from the modified Saltzman procedure of Levaggi, *et al.*³ Nitrogen dioxide sidestream deliveries are given for several cigarettes in Table 1.

TABLE I
NITROGEN DIOXIDE IN SIDESTREAM SMOKE

Cigarette Code	Description	NO ₂ (μg/cigt)
X6DOECE	Ca(NO ₃) ₂ on MF (9.97% NO ₃ ⁻)	4215
X6DOECF	Mg(NO ₃) ₂ on MF (6.83% NO ₃ ⁻)	2949
X6DOECD	NaNO ₃ on MF (8.15% NO ₃ ⁻)	874
X6DOECC	KNO ₃ on MF (7.97% NO ₃ ⁻)	139
D2CUS	Schweitzer SBS, Uncased	192
X5D5UU	All Burley	112
X5D5UW	All Turkish	34
X5D5UV	All Bright	33
Ky Ref 1R1		26
6169-78	Cellulose Base, + NaNO ₃ (0.6% N)	25
JABQ-3AT	Cellulose Base, + Knox Gelatin (2.1% N)	16

2022158636

The gross differences between deliveries from cigarettes which contained calcium and magnesium nitrates and the other nitrate-treated cigarettes are consistent with observations made by other workers concerning alkali and alkaline earth nitrate behavior.^{4,5,6} In the cited works, calcium and magnesium nitrates readily decompose on heating in air to give nitrogen oxides and metal oxides whereas the sodium and potassium nitrates decompose to oxygen and nitrites which are very stable to heat.

Sidestream to mainstream ratios of nitric oxide deliveries $\left[\frac{(SS)}{(MS)} NO \right]$ have been found to be inversely proportional to nitrate concentration in filler. A plot of $\log \left(\frac{(SS)}{(MS)} NO \right)$ versus the log of percent NO_3 for several cigarettes yields a straight line of negative slope. These data agree with results previously observed for nitrous oxide and further support the hypothesis that the importance of the nitrate decomposition mechanism, relative to the organic nitrogen oxidation mechanism, is greater in the formation of the mainstream.

A perusal of mainstream nitric oxide deliveries of commercial cigarettes shows Viceroy > Marlboro > Kent and Winston. Sidestream nitric oxide was previously found to be highest in Marlboro.

II. KETENE IN CIGARETTE MAINSTREAM SMOKE⁷

Experimental cigarettes were smoked and ketene was measured as the n-butyl acetamide derivative along with phenol and dimethyl phenol. These data are summarized in Table II.

TABLE II
KETENE AND PHENOLS IN CIGARETTE MAINSTREAM SMOKE

Cigarette	Description*	Ketene (μ g/cigt)	Phenol (mm Peak Height)	Dimethyl Phenol
6169-79	α -Cellulose	18	43	18
6169-86	α -Cellulose, 2-Deoxyglucose	36	63	175
6169-88	α -Cellulose, Silver Acetate	2.2	2	1
6169-89	α -Cellulose, Silver Acetate, 2-Deoxyglucose	20.4	21	122

* To regulate burn, cigarettes contained tricalcium phosphate and potassium chloride.

2022158637

Ketene yields follow predictions based on pyrolytic studies in that 2-deoxyglucose was shown to be an efficient ketene producer at several temperatures. The dramatic reduction in ketene by silver is of great interest in that it is to be expected as a consequence of the formation of silver ketenide $\text{Ag} \begin{array}{l} \diagup \\ \diagdown \end{array} \text{C}=\text{C}=\text{O}$. We are seeking structural

evidence for this compound but have no instrumental data in hand. However, the ketenide is reported to be yellow.⁸ The ash of cigarette #6169-88 is yellow and loses its color slowly on standing.

It was also noted that phenol yields varied in the same directions as did ketene yields. The role that ketene may play in phenol formation is under study.

III. PYROLYSIS OF CITRIC ACID AND SALTS THEREOF⁷

An apparent relationship exists between ketene formation efficiency and acetonitrile formation when ammonia is added to the pyrolysis system in the form of ammonium carbonate. (Table III).

TABLE III

PYROLYSIS OF CITRIC ACID AND SALTS THEREOF		
Compound Pyrolyzed (0.1 mmol)	Ketene (μg)	Acetonitrile* when 0.1 mmol $(\text{NH}_4)_2\text{CO}_3$ Added
Citric Acid	184	140
Ferric Citrate	154	24
Calcium Citrate	114	13
Potassium Citrate	54	6

*In recorder peak height

These results raise the possibility that some salt casing action may be a consequence of changes in the efficiency with which ketene is produced. This investigation is continuing.

2022158638

IV. REFERENCES

1. R. W. Hale, Notebook 6214, p. 24-31.
2. D. H. Powell, Notebook 6097, p. 53-65.
3. D. A. Levaggi, et al., J. Air Pollution Control Assoc., 33(1) 30-33 (1973).
4. C. Duval, "Inorganic Thermogravimetric Analysis," 2nd ed, Elsevier, N.Y., 1963, p. 270.
5. W. Wendlandt, Texas Journal of Science, 10, 392 (1958).
6. W. Johnson, Technical Report 291, April, 1970.
7. J. C. Kang, Notebook 6203, p. 44-59.
8. R. Lewin, New Scientist, December 21, 1972.

/mro



2022158639

CHARGE NUMBER: 1300
PROGRAM TITLE: Sheet Materials Process
PROJECT LEADER: G. Gellatly
PERIOD COVERED: May 1 - 31, 1973
DATE OF REPORT: June 12, 1973

I. RCB

A production run using coarse-ground burley stems was carried out. The objective was to eliminate the drying and processing cost of the burley stems prior to milling and screening. In this run the stems were milled at 12% O.V. and were not screened prior to blending. The tensile strength of the sheet was not significantly different from regular RCB in the longitudinal machine direction but some 12% lower in the transverse direction.¹ Cigarettes made from this material will be tested against control RCB before this mode of operation is considered for adoption.

Subjective smoking data for cigarettes containing RCB with 41%, 44% and 51% burley stems is not yet available.

II. Schweitzer Sheet

The third production run this year (No. 3-73) of burley stem sheet was made May 1, 1973 to May 10, 1973. The overall yield was 84%. The % solubles reconstitution was again low (76.4%, spec. 85%). The average % TEG was within specification (1.9%, spec. 1.75% to 2.25%). No potassium sorbate was found in 18 of the 26 samples received. A recommendation was made to Schweitzer that they increase their potassium sorbate addition to 0.25%/lb dry tobacco on their next production run to compensate for losses in drying. The first eight samples of this run were found to average 0.20% (specification - 0.1% to 0.2%).²

The lack of potassium sorbate in SBS - 3-73 is of real concern. A sample of SBS was received from Louisville which had no potassium sorbate and was molded. The moisture content of this sample was 21.4%. The equilibrium moisture content of SBS was determined to be 23.5% at 90°F and 85% RH.³ Under

2022158640

these typical summer conditions, Manufacturing will have to be vigilant for molded SBS. The Leaf Department was advised to use this last production run (3-73) before previous runs to minimize time for molding.

Work has begun to find the optimum method for effluent disposal from the proposed Schweitzer pilot plant for R&D. We are establishing the BOD₅ of their effluent and have enlisted Calgon Corporation's help to determine applicability of activated carbon to lower the BOD.

Caramel color has been proposed to improve the color of SBS, and cigarettes containing 2% SBS with caramel color will be evaluated subjectively.

Burley spray take-up on SBS relative to burley strip is being investigated. Initial results show SBS to contain 30% more sugar than burley strip after spraying.⁴ Further samples are being analyzed.

III. Nitrate Reduction of Burley Stems

Subjective evaluation of cigarettes containing 25% and 95% nitrate reduction are awaited. The nitrate and solubles were reduced by conventional washing and by pressing wetted stems. The pressing method is advantageous because it would reduce the drying cost by ~60% and also reduce the quantity of effluent to be handled by 80%. A production trial using a sugar mill as a press is proposed to evaluate this equipment's applicability to tobacco processing.

IV. References

1. Notebook 6099, pp. 66 - 69.
2. Notebook 5588, p. 72.
3. Notebook 6168, p. 53.
4. Notebook 6168, p. 54.

/jj


G. Gellatly

2022158641

CHARGE NUMBER: 1503
PROGRAM TITLE: Modified Smoking Materials
PROJECT LEADER: J. D. Hind
PERIOD COVERED: May 1 - 31, 1973
DATE OF REPORT: June 14, 1973

I. Film Wrappers¹

Another batch of 18,000 film-wrapped cigarettes was made this month. They will be fitted with several different types of filters and used in a continuing study of attachment adhesives.

Modification of the doctor blade angle (at the Sandvik belt) has permitted us to make straight, tight, nearly perfect parent rolls of film using the surface winder. The excellent performance of the surface winder may be partly due to the fact that the winding is conducted in a reverse direction to the natural curl of our incompletely equilibrated films.

The Descriptive Panel confirms the cigar-like character of the unventilated "film" cigarette (Marlboro 85 mode) and rated it one unit higher on the hedonic scale than Marlboro standard in 15 of 23 attributes.

II. Designed Fillers²

We varied our standard ammonium pectate formulation separately with 0.03% or less valeric, isobutyric acids, 3% corn and potato starch, 0.3 and 0.15% of three sulfur amino acids and 3% pectin. The sulfur amino acids contribute both favorable and unfavorable character and must be used at extremely low levels. The other materials contributed satisfactory characters to the smoke at the above levels.

A specially prepared ammonium alginate (by Kelco in their laboratory) was close in aroma character to the demethylated pectin we have been using. Present plans include preparing formulations made with calcium pectates suspended in low viscosity guar gum binders.

2022158642

Burley and bright strip paraffin waxes have been characterized by infrared spectrograms before and after treatments in warm concentrated H_2SO_4 (G. Vilcins, Notebook 6283, pp. 77, 83). The purpose in obtaining this data is to facilitate substitutions of leaf paraffin by petroleum paraffins in future experiments.

We are also working with several lipid emulsion formulations which will be used at below 1% levels in RKS and shredded stem to control mildness.⁴ Lipids are not expected particularly to reduce stemmy pyrolysis character.

III. Shredded Stems³

Materials have been prepared for a direct comparison of the Fitzmill shredding and the RKS process. The comparison will include measurements of filling power, cylinder volume, analytical and subjective evaluations on washed and unwashed, expanded and unexpanded bright and burley stem fillers made by both processes from split samples of starting material.

Appropriate measurements are being made on the initial "demonstration" run (Fitzmill Process) cigarettes made last month. Evaluations by the Flavor Group indicate that partial extraction of water solubles makes a moderate improvement--that addition of the special casing contributes an even larger smoke improvement.

(The Fitzmill process treatment eliminates the steps of rolling and cutting and has been shown to give comparatively high cylinder volumes.)

IV. References

1. E. J. Deszyck - Notebook 6222, pp. 58 - 74.
2. J. W. Leik - Notebook 6233, pp. 23 - 35.
3. G. F. Kite - Notebook 6278, pp. 22 - 37.
4. J. D. Hind - Notebook 5273, pp. 91 - 94.



J. D. Hind

/jj

2022158643

CHARGE NUMBER: 1504
PROGRAM TITLE: Designed Filler
PROJECT LEADER: G. D. Keritsis
PERIOD COVERED: May 1 - 31, 1973
DATE OF REPORT: June 12, 1973

I. Low Tar Filler (LTF)

1. The 1972 LTF sample requested by Chemical Research was given to Cigarette Development Group in filler form.¹
2. The evaluation of various 1973 experimental cigarette models continued.²

II. Natural Base Filler (NBF)

1. Two large batches of lemon albedo, product C-106, were ozonated in a large glass column.³ This material produced a designed filler slurry of an adequate viscosity, and it was easily converted into an acceptable designed filler sheet.⁴

The sheet was cut into small squares and blended with tobacco at the 50% level. Flavors will be applied, and the machine made cigarettes will be evaluated by Lab and POL panels.

2. A medium size extractor was obtained, and one pound quantities of coffee bean hulls are being extracted. This material will be used as a designed filler base in a larger evaluation.

III. D.F. Processing

1. Sheeting

Continuous and strong designed filler sheets can be made at the R&D and BL pilot plant facilities.

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2. Conditioning

Proper cutting moistures (12 - 16% O.V.) can be achieved in a manner similar to conditioning ET (Proctor & Schwartz reordering unit).⁵

3. Cutting

The designed filler or any other smoking material (tobacco) in sheet form can successfully be cut into an acceptable cigarette filler on the Molins cutter providing a uniform feed rate is maintained. Mechanical means of continuously and uniformly feeding the cutter are being examined.

4. Cigarette Making

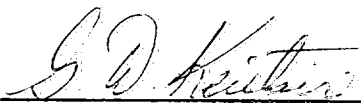
The cigarette making of 100% D.F. or reconstituted tobacco on the Mark VIII is indeed a very troublesome process. Proper feeding of the filler in the chimney section of the making machine hopper, and the ecretuer section which gets jammed up with the trimmed recycled material are two of the most critical and troublesome sections of this unit.

Recommendations were made⁶ to (a) internally reduce the tensile strength and tear characteristics of designed filler and (b) reduce the size of the cut material to a strand length between 1/4 to 3/4 inches. Means to comply with these recommendations are now being evaluated.

Approaching this problem from a different direction, George Mathe has agreed to explore modifications to the cutting and making equipment to adapt it for 100% D.F. materials.

IV. References

1. Notebook 6169, p. 95.
2. Notebook 6169, pp. 96 - 100.
Notebook 6315, pp. 1 - 2, 5 - 7.
3. Notebook 6119, p. 63.
4. Notebook 6240, pp. 42 - 47.
5. Notebook 6315, p. 4.
6. Memo: "LTF-Mark VIII Machinability" to G. D. Keritsis from L. L. Long, dated May 24, 1973.


G. D. Keritsis

/jj

2022158645

CHARGE NUMBER: 1600

PROGRAM TITLE: Smoker Psychology

PROJECT LEADER: W. L. Dunn, Jr.

PERIOD COVERED: May 1 - May 31, 1973

DATE OF REPORT: June 5, 1973

Project Title: Smoking and Rate of Learning Alpha Control
Written by: W. L. Dunn

With several preliminary sessions on the equipment, the writer has affirmed that the alpha learning phenomena is real. An auxiliary circuit is being added to the equipment for recording cumulative time on target. When readied, more systematic observations will be begun.

Project Title: The Delivery of Inhalation Impact via Vehicles Other Than Nicotine
Written by: W. L. Dunn

We smoked amongst ourselves some partially (80%?) denicotinized 100% burley cigarettes hoping that there would be residual impact. There was virtually none. Had there been, we would have wanted to exploit this characteristic by designing it into denicotinized cigarettes in order to determine whether smokers smoke for impact per se or use impact as a cue for the presence of nicotine. The project is discontinued until a means for delivering inhalation impact other than via nicotine is available.

Project Title: Another Dilution Control Center for the Smoker
Written by: W. L. Dunn

Still in very preliminary stages.

Project Title: Arousal & Smoking
Written by: Frank Ryan

Data gathering continues.

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Project Title: Cigarette Variability
Written by: Frank Ryan

Cigarettes are now on hand with average delivery just under 11 mg tar. Pack handouts will be made in June to low delivery smokers of the Richmond Product Placement Panel.

Project Title: CIG-1
Written by: Frank Ryan

Data gathering is underway using local college student smokers who come to the Behavioral Research Lab. Depending on subject availability data should be complete in July.

Smokers take 20 puffs at about one minute intervals, receiving a newly lit fresh cigarette after each two puffs. We are looking for back-off effects on puff volume and duration.

Project Title: Methods Studies
Written by: Frank Ryan

In two separate methods study tests we have failed to find any presentation procedure which seems more sensitive than any other procedure. When differences in cigarettes were very slight, all methods used showed chance results. When differences were fairly large, all methods used showed significant or near significant differences between products. Next comparison test scheduled is for a moderate difference.

Project Title: Acceptability and Low Delivery Cigarettes (TNT-3)
Written by: T. R. Schori

Analysis of these data should commence shortly by John Tindall.

Project Title: Smoking and Spare Mental Capacity
Written by: T. R. Schori

Including pilot subjects, a total of 55 college students have been run in this study so far. One thing is certain: unlike many psychological studies, the students have not found this study monotonous.

Project Title: Tar, Nicotine, and Smoking Behavior (TNT-4)
Written by: T. R. Schori

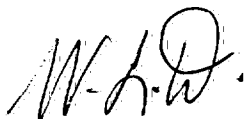
Cigarettes for the fourth week have just been sent out to the RP³ panelists participating in this eight-week study. Thus far the study seems to be running reasonably smoothly.

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Project Title: Nicotine Optimazation in Menthol Cigarettes
(MN-2)

Written by: T. R. Schori

This study was designed to identify nicotine delivery levels which might feasibly be utilized in Alpine-like 85 mm menthol cigarettes. Six nicotine deliveries were tested on 70 POL/SEF panelists - 1.09, 1.35, 1.54, 1.79, 1.86, and 2.08 mg. Preliminary data analysis indicates that the lowest delivery was most acceptable with acceptability declining with increasing nicotine. A brief report is being written.



/j1h

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CHARGE NUMBER: 1706
PROJECT TITLE: TOBACCO PHYSICS
PERIOD COVERED: May 1 - May 31, 1973
DATE OF REPORT: June 4, 1973
WRITTEN BY: A. C. Lilly

A. Conversion of Rod to Smoke^(1,2)

Work has continued on the addition of the solid phase equations to general combustion program. At present we feel that the weight loss, condensation, and CO, CO₂ production can be written in kinetic form. The CO and CO₂ production from tobacco as a function of temperature is being fit to two and four one-step kinetic expressions respectively. The temperature dependent part of the condensation is being derived from the weight loss function minus the CO, CO₂ at each temperature. The spatial dependence of the condensation function has been derived from recent data reported by Palmer. (3) Palmer's data indicates an apparent increase in TPM production of a burning cigarette appears to be widely distinct from a steady-state process. Indications are that 25-35 per cent of the TPM produced in a puff is deposited in the first 6.5 mm of rod behind the coal, 10-20% in the second 6.5 mm, and negligible amounts thereafter.

The new variable parameter smoking machine has been constructed and is presently being checked and calibrated. Shortly, we will begin obtaining data on cigarette TPM output and filter efficiencies as a function of flow rate.

A combination smoking machine and gas sampling system has been constructed for the purpose of sampling the gas phase as a function of distance behind the coal in a burning cigarette. A gas chromatographic technique is being used to measure the pertinent gases (i.e. N₂, CO, CO₂, and O₂). (4)

B. Catalysis Mechanism Studies⁽²⁾

The reflectance and transmittance cell compartments of the new Beckman DK-1 spectrophotometer have both been checked and appear to be satisfactory. All detectors are operating as specified. Reflectance spectra of supported metal oxides are in agreement with previous work. The transmittance attachment will be used to carry out in situ NO analysis of synthetic gas mixture exposed to potential NO active filter materials.

The spectra scan 750 Residual Gas Analyzer has been set up to investigate the CO on Al₂O₃ and the permanganate catalysts, hopefully

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to determine the basis for their catalytic activity. The common power supply for the ion energy and the electron collector, voltages was found to be faulty and has been corrected. The Keithley 416 picoammeter was not operating correctly and was sent to the factory for repair. Modifications of the vacuum system, are being made to allow evacuation of the catalyst material through an activated charcoal trap in order to collect gases given off by the catalyst during evacuation. (5)

C. Miscellaneous (6)

Sensor substrates are being made to send to the fourteen companies actively interested in licensing the technology.

/gdc

References:

- (1) A. C. Lilly, Book 6292, pp. 1-10.
- (2) Allen Kassman, Book 6277, pp. 25-33.
- (3) A. M. Palmer, Special Report No. 73-060, April 27, 1973.
- (4) H. V. Lanzillotti, Book 5584, pp. 62-63.
- (5) J. C. Crump, Book 5411, pp. 86-88.
- (6) C. O. Tiller, Book 6148, pp. 49-58.



A. C. Lilly

2022158650

PROJECT NUMBER: 1708
PROJECT TITLE: FILTER MATERIALS RESEARCH
PROJECT LEADER: N. B. Rainer
PERIOD COVERED: May 1 - May 31, 1973

Cigarette-like smoking articles consisting entirely of carbon (except for the wrap paper) have been made by pyrolyzing excelsior in the form of a rope-like structure. The 85 mm length "cigarettes," which provide 10 puffs in a standard smoking test, deliver essentially zero TPM, only 6 mg CO, and extremely little gas phase organics. (1) This structure serves as a useful model for our studies of the origin of CO in cigarette smoke and for investigations of flavor transfer and effects of the cigarette wrap on delivered smoke.

Small diameter carbon rods have been made by the pyrolysis of strands of sisal twine. These have been used for making core-type cigarettes. (2)

A number of samples of lemon albedo have been treated with NO₂ and with combinations of ozone and ammonia. (3) The resulting materials have been submitted to W. C. Hopkins for evaluation. It has been found that, once treated with NO₂, lemon albedo continues to reactively degrade until extracted.

Two large batches of lemon albedo (3 kilos each) have been treated with ozone. (4) These are to be converted to cigarettes for subjective evaluation. Apparatus to facilitate gas treatment of cigarette paper has been designed and is presently being fabricated in the shop.

/gdc

References:

1. Notebook 6281, pp. 54, 55, 69.
2. Notebook 6281, pp. 60-64, 75-78, 85.
3. Notebook 6119-A, pp. 64-66.
4. Notebook 6119, p. 63.

N. B. Rainer

2022158651

CHARGE NUMBER: 1801
PROGRAM TITLE: Tobacco Processing
PROJECT LEADER: F. V. Utsch
PERIOD COVERED: May 1 - 31, 1973
DATE OF REPORT: June 13, 1973

I. Impregnation Studies

A. Water Impregnation

Experiments using five different detergent types to aid penetration of the water into the tobacco confirm that detergents are helpful in achieving expansion at low water levels, but are detrimental to expansion at the higher water levels. Studies will be continued to further define the best class of detergents and to define which of the tobacco components have the greatest resistance to water penetration. Use of towers for expanding the treated filler is also being attempted.

B. Expansion with CO₂

Test runs were conducted at Airco to establish nominal operating ranges and pilot equipment requirements. It was concluded that liquid CO₂ is necessary to achieve suitable impregnation. Pressures of 500 - 600 psig were required to obtain penetration into the tobacco with no apparent advantage to using pressures over 600 psig. The time required to achieve impregnation is five minutes or less, and the permissible hold time after impregnation is very dependent on temperature.

A pilot plant process is now being defined and plans are to begin ordering equipment this month.

II. Tobacco Drying Process Studies

A. Jetstream and Closed-Loop Pilot Adt Dryers

Cigarettes made at a 7% weight reduction and designed by the Smoke Filtration Group to give tar deliveries equal to the standard Marlboro control sample have been smoked

2022158652

by the Flavor Development Group, and no significant differences were found.

These results indicate that previous subjective differences detected for the experimental cigarettes may have been caused by unequal deliveries and not by steam atmosphere processing of the filler. Larger scale subjective testing is recommended.

The Jetstream dryer has been relocated to the new "CCD" pilot plant. Filler drying at 4,000#/hr will be evaluated to determine equipment scale-up and commercial feasibility for the unit as a dryer for cut filler.

B. Tobacco Dryer Fundamental Studies

Filling power increase of MF blend was found to be in direct proportion to the steam concentration in the process atmosphere of the pilot Adt dryer over a range of 2% - 98% steam. A decrease in the equilibration moisture levels of the treated filler was also observed to be in direct relation to increasing steam concentration. The cause of this decrease is not known. Further studies of the filler steaming relationships are currently underway in the Mohr and Aminco units.

Good agreement with published relationships and good data reproducibility are being obtained in the study of dryer operating parameters versus retention time and conveying in the pilot Adt dryer. A wide range of dryer RPM, angle, feed rate and gas flows are being evaluated. Correlation of drying efficiencies and product filling power versus dryer operating parameters are also being attempted. A systematic review of chemical abstracts for literature related to heat transfer in rotary dryers is underway. Two drying consultants are scheduled to visit in June to assist in program definition and analysis of experimental results.

Initial drying curve studies have been completed and show that below 50% O.V., tobacco is in the falling rate period of drying.

2022158653

III. References

1. Notebook 6301, pp. 7 - 45.
2. Notebook 6072, pp. 75 - 85.
3. Notebook 6086, pp. 98 - 99.
4. Notebook 6303, pp. 1 - 13.
5. Notebook 5518, pp. 42 - 62.

F. V. Utsch

F. V. Utsch

/jj

2022158654

CHARGE NUMBER: 1802
PROGRAM TITLE: Expanded Stems
PROJECT LEADER: L. R. Turano
WRITTEN BY: J. M. Whitman
PERIOD COVERED: May 5 - June 5, 1973
DATE OF REPORT: June 12, 1973

I. Westab Expanded Stem Facility

Buildup of stem material occurs at both the inlet feed section and the first elbow. The 18" plexiglass tower is being installed in the R&D pilot plant to study stem flow patterns and evaluate the use of different deflectors to alter the flow and prevent the buildup.

II. Swiss Expanded Stem Facility

The expansion level of the Swiss product began steadily decreasing during the week of May 28. The cause of this problem could not be determined by the Swiss. Messrs. Turano and Knight have returned to Switzerland to investigate.

III. Comparison of Westab and R&D Towers

A program is underway to compare the products from the Westab and R&D towers using the same starting batches of:

1. Long, bright stems.
2. Blend of 67% long, 33% short, bright stems.
3. Bright cased filler.

The test on long, bright stems has been completed. Data are given below:

2022158655

<u>Tower</u>	<u>% O.V.</u>	<u>Reducing*</u> <u>Sugar</u>	<u>Total*</u> <u>Alkaloids</u>	<u>C.V.</u>	<u>% O.V.</u>
R&D	8.5	11.5	0.48	47.1	13.3
Westab	11.6	11.6	0.49	45.3	13.1

* Dry Weight Basis

Cigarettes made with each product are being made by the
Total Utilization Group.


J. M. Whitman

/jj

2022158656

CHARGE NUMBER: 1901
PROGRAM TITLE: BIOCHEMICAL MODIFICATION OF TOBACCO
PERIOD COVERED: May 1 - May 31, 1973
PROJECT LEADER: D. M. Teng
DATE OF REPORT: June 1, 1973

I. BIOCHEMICAL MODIFICATION OF TOBACCO

A. Artificial Flue-Curing¹

Artificial flue-curing of greenhouse-grown tobacco was continued to compare with farm curing. At the end of 96 hours, invertase activity was 235% of the normal control and the catalase activity was 277% of the normal control. The difference in β -amylase activity was not significant. These findings will be verified by experiments with field-grown leaves in the summer.

B. Electrophoresis of Protein²

Experimental data indicate that cellulose acetate is not the ideal support media for the separation of lipo protein in tobacco.

Agarose-gel film will be tried for this separation.

II. BEETLE EXTERMINATION PROGRAM³

Insects and observations were provided for eleven "experimental runs" during the month of May. No serious problems have been encountered in doubling the number of insects and observations supplied to Dr. T. Laszlo.

III. REFERENCES

1. D. Teng 6273
2. B. Semp 6185
3. B. Lehman 6312

W. R. McCoy

/slb

2022158657

CHARGE NUMBER: 2100
PROJECT TITLE: SMOKE FILTRATION
PERIOD COVERED: MAY 1 - MAY 31, 1973
PROJECT LEADER: P. N. GAUVIN
WRITTEN BY: W. G. HOUCK AND L. F. FILOSA
DATE OF REPORT: JUNE 12, 1973

I. NEW CIGARET DEVELOPMENT

A. Parliament 85/80 Filter Modification

The lengthened filter on the Parliament CFF blend with compensation is currently being RP³ and HTI tested in the 85 mm configuration. The 80 mm models are currently being assembled for HTI testing.

B. 85 mm Slim Cigaret Development

Tar delivery specifications and internal subjective testing have been completed on both the experimental 85 mm slim and a standard circumference control.

We are currently awaiting special box packages from American Can Co. Once these are received, the boxes will be hand packed and RP³ testing will commence.

C. Exposed Plastic 100

Hand-made samples of the 100 mm model showed the tar delivery to be higher (23 mg) than would be acceptable for mailout testing. A new tow item has been ordered that should reduce the delivery to the desired level (18 mg).¹

In the meantime, trial maxing runs with the reduced roughness foamed plastic filter have been scheduled.

D. Active Carbon Menthol Cigaret

Initial testing shows that with the use of special starch based spray-dried menthol particles, an active carbon menthol cigaret can be achieved. These particles are located on the CA plug behind (mouth end) the carbon on tow section. Flavor transfer is occurring via the particle fallout mechanism.

The particles do not appear to release a significant amount of menthol to the carbon during aging, therefore not effecting the activity of the carbon or the menthol delivery.²

Further analytical and taste evaluations are planned with machine-made models.

2022158658

E. 12 mg Full Flavor Cigaret Development

A new program to develop a 12 mg full flavored 85 mm cigarette has been initiated. Prototype models utilizing four different blends and two types of filter systems have been designed and fabricated. The Flavor Group will screen these samples for the more acceptable models. Further blend and/or filter modifications will be made if necessary.³

The initial subjective and analytical evaluations should be completed by the end of June, 1973.

F. Low Delivery Cigaret With Dry Flavor Particles In The Filter

Taste improvement of low delivery cigarettes in the 5-6 mg and 10-12 mg tar ranges containing distinctive flavor particles in the filter are being investigated with the Flavor Development Group. Flavor particles with a tobacco-like response will also be evaluated at these tar levels. Currently, distinctive flavors at 10-12 mg tar are under evaluation.

II. CIGARET COMPONENT DEVELOPMENT

A. Porous Tipping

A POL mailout of a Marlboro 100 type cigarette utilizing the Ecusta cork, porous tipping versus a Hauni diluted Marlboro 100 has been initiated.

Internal subjective testing found no differences between a standard Marlboro Lights and a Marlboro Lights with Ecusta white, porous tipping. Mailout testing is planned when the specially printed white, porous tipping arrives from Ecusta. The printing is needed in order to conceal the rod-filter juncture.⁴

Samples of French (Malaucene) and German (Benkert) cork, porous tipings have been requested for our evaluation.

B. Porous Filter Rods

Breadboard equipment to attempt the post forming of grooved Sphero-pore A (polyurethane) rods from a flat sheet was delivered for evaluation. An investigation of the possible extrusion of Sphero-pore A is still awaiting necessary equipment.

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Samples of a new porous polymer, Sphero-pore B, were received from Monsanto. An initial evaluation indicated Sphero-pore B to be equivalent to Sphero-pore A in TPM efficiency and taste. Physically, the samples were too powdery and friable and, thus, unacceptable. Monsanto is aware of these problems and is attempting to improve the product.

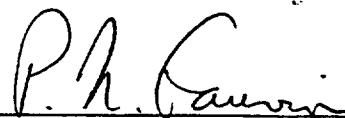
C. Nitrogen Oxides Removal in Smoke

An aging evaluation is continuing on several permanganate samples utilizing different base materials. Several indicated 55-70% NO removal in smoke with fresh cigarettes.⁵ Subjective evaluation will be initiated on those samples retaining significant NO activity after aging.

Further investigation of 5% Salcomine (cobalt complex) on carbon in a P-S-P filter for NO removal will be discontinued. It has been analytically determined that cobalt or a derivative is detected in mainstream smoke due to Salcomine-coated carbon particle fallout.⁶ Attempts to eliminate detectable cobalt fallout by using a carbon with low dust-off and a high denier CA back-up were unsuccessful. A Marlboro control cigaret indicated no detectable cobalt in mainstream smoke.

REFERENCES

- 1 - Notebook 6167, p. 97.
- 2 - Notebook 6227, pp. 50, 53, 63, & 66.
- 3 - Memo, P. Gauvin and C. Kounnas to J. Osmalov and F. Daylor, 3/20/73.
- 4 - POL Booth Test Data, 5/29/73.
- 5 - Notebook 6316, p. 2.
- 6 - Memo, W. R. Morgan to L. F. Filosa, 5/29/73.


P. N. Gauvin

/gmm

2022158660

CHARGE NUMBER: 2105

PROJECT TITLE: FILTER & CIGARET PROCESS DEVELOPMENT

PERIOD COVERED: MAY 1 - MAY 31, 1973

PROJECT LEADER: J. M. WASHINGTON

WRITTEN BY: J. F. NIENOW & J. M. WASHINGTON

DATE OF REPORT: JUNE 12, 1973

I. EXTRUSION SYSTEMS DEVELOPMENT

Mechanical and electrical modifications of #1 Extrusion Line have been completed. Extended reliability runs producing 90 mm "Multifilter" plugs at 500 fpm will be made prior to a demonstration run for Manufacturing management, scheduled mid-June.⁽¹⁾ If accepted by Manufacturing, the line will be disassembled and shipped to Louisville.

Extrusion Line #2 will be reactivated in place to provide extrusion capability. Mechanical and electrical rework in the location now occupied by #1 Line will include evaluating a Molins bed (garniture and cutter) in addition to a standard puller-cutter assembly. A schedule for Line #2 renovation has been issued.⁽²⁾

II. FFM-5 PRODUCT DEVELOPMENT

Work has continued, evaluating adhesives and papers, to produce an acceptable flush-fluted single filter cigaret with blocked flutes and reduced flute definition on tipping paper.⁽³⁾ A hot melt system for blocking flutes and/or sealing tipping paper has been proposed by Mercer Co.⁽⁴⁾ and will be investigated.

III. SCULPTURED FILTER DEVELOPMENT

Final modifications of the breadboard unit will be made for product demonstration. A design review was held on the prototype off-line sculpturing unit, and revisions of the design have been started.

2022158661

IV. POWDER ON TOW APPLICATOR

A Molins plugmaker has been modified to provide spatial flexibility for installation of various powder applicators. Mechanical and electrical work on the basic machine should be completed by July 1. A brush-screen powder applicator design was reviewed and has been released for fabrication.

V. GRANULAR SPACE FILLING DEVELOPMENT

A Baumgartner vacuum-assisted space-filling unit was received, and installation on the Molins P-S-P machine has begun. Damaged parts were returned to Baumgartner for repair or replacement, and information on the heat-sealed combining wrap used on this device has been requested.

VI. "SPHERO-PORE A" FILTER PROCESS DEVELOPMENT

Mixing and metering equipment for pilot scale demonstration of "Sphero-pore A" production has been ordered. Receipt of this equipment will provide capability of extruding rods of filter material. Post forming "Sphero-pore A" is being investigated by L. Filosa. Shaped molds will be ordered upon receipt of a second price quotation.

VII. TOBACCO DUST RECLAMATION BY EXTRUSION

Scouting studies were initiated to determine feasibility of extruding tobacco dust. Several feed stocks were extruded using the capillary rheometer, and 0.020" strands with relatively low tensile strength were produced.⁽⁵⁾

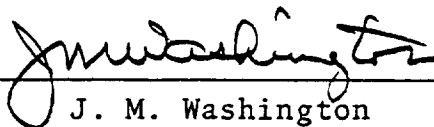
VIII. RING TIPPING PROCESS DEVELOPMENT

A short series of tests was run to determine parameters for ring tipping exposed plastic filters to cigarettes.⁽⁶⁾ An interim process was defined, but further development is anticipated to demonstrate acceptable performance at manufacturing speed.

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REFERENCES

- (1) Memo: Mr. F. E. Resnik, "Extrusion Line for Louisville," June 8, 1973.
- (2) Memo: Robert Gaudlitz to Mr. W. F. Mutter, "Extruder Reconstruction Schedule," May 30, 1973.
- (3) A. Gergely - Research Notebook 4674, pp. 75-80.
- (4) Letters: M. F. Mercer to J. F. Nienow, April 9, 1973, and May 23, 1973.
- (5) J. F. Nienow - Research Notebook 5501, pp. 30-32.
- (6) A. Gergely - Research Notebook 4674, p. 74.


J. M. Washington

/gmm

2022158663

CHARGE NUMBER: Various
PROGRAM TITLE: Flavor Development
PERIOD COVERED: May 1 - 31, 1973
PROJECT LEADER: H. L. Spielberg
DATE OF REPORT: June 11, 1973

I. FLAVOR PRODUCT DEVELOPMENT

Test marketing of a CF free Parliament with flavor compensation has been authorized. Materials for flavor 5367-6 have been ordered and will be transferred to the Manufacturing Department after they have been screened. CF replacement work on Multifilter and Marlboro Lights is continuing.¹

Work with reduced delivery cigarettes, including 12 mg and 14 mg models is proceeding. Two of the three 14 mg Hi Burley models were below target delivery. These lower delivery models will be screened as 12 mg candidates and also be remade at the higher delivery level. A series of five blends with two different filter constructions are being prepared as 12 mg candidates.²

Several cigarette models in the menthol program are currently being evaluated. Marlboro Lights-type menthol models have been boothe tested against Salem and Kools. Richmond placement testing of this cigarette is scheduled. Preliminary manufacturing specification runs for Alpine 100 have been completed and specifications are being written. Two National POL mailout tests of modified Alpine vs. regular Alpine have been released and the HTI test is being analyzed. Preliminary manufacturing specifications of Marlboro 80 Menthol are being established. Models are also being prepared for consumer testing. Marlboro-type cigarettes, with a low level of menthol have been prepared and preliminary testing are being conducted. Boothe testing of this model showed us obvious differences from the control.³

In the utilization studies, modified flavor systems have been developed for use on ESS based on the 15% prototypes previously evaluated. The flavor systems were developed for national testing of ESS at about 7%. Additional work is being conducted to develop flavor systems for ESS and ES that are more easily adaptable to current processing procedures. July 1973 type models are being prepared with cased and uncased ES as well as regular RKS, for extensive evaluation. Cylinder volumes for the three flavored ESS samples were found to be comparable with the unflavored ESS. Flavor development work on EBS as well as ESS has included the development and evaluation of reducing sugars, dap, ammonia, and some suspected flavor precursors such as sulfur-amino acids. Additional amino acid, sugar combinations are being evaluated, alone and in combination with conventional flavors. Studies of SRT and SBS are continuing. Models of cigarettes with flavored SRT at 5%, 10%, and 17 1/2% are being evaluated. National POL models using 22 1/2%

2022158664

TBRCB are in cold storage awaiting an available panel. STBRT is being tested by HTI in cigaret models at 22 1/2%. Preliminary data show this model to be different from the control.⁴

Flavor modified Parliament cigaretts have been prepared and are being evaluated. HTI models of Parliament with filter modifications have been screened subjectively and found satisfactory for release.⁵

II. FLAVOR DEVELOPMENT ASSISTANCE

A series of flavors have been developed and applied to NBF products and LTF material. Cigaretts using these flavored materials at the 50% level are being evaluated.⁶ RGL cigaret models are being screened and flavor evaluated on this product.⁷

Turkish casing flavors were screened for use in Venezuela. Flavor 6218-52B was recommended for further evaluation.⁸

Evaluations of cigaret models with modified sidestream are underway as well as the development of flavors for addition to the cigaret paper. Work on distinctive flavors and flavor fall out is also continuing.

III. REFERENCES

1. Notebook 6205, page 83.
" 5367, page 6.
2. " 6205, pages 72, 84.
" 5367, page 20.
3. " 6205, pages 70-71, 73-74, 76-77, 79, 81-82, 89.
" 5367, page 16.
4. " 6277, pages 98-100.
" 6321, pages 1-35.
" 6295, pages 10, 12-13, 26, 27.

Memo L. L. Long to D. K. Keel, May 17, 1973. "Cylinder Volume of Flavor Treated ESS."

Notebook 6293, page 23.

5. " 6293, pages 27-28, 34, 40.
6. " 6293, pages 17, 21, 25, 29, 35, 36, 37.
7. " 6293, page 38.
8. " 6293, page 48.

/wt


H. L. Spielberg

CHARGE NUMBER : 2306
PROGRAM TITLE : Flavor Transfer
PROJECT LEADER : R. M. Ikeda
PERIOD COVERED : April 26 - May 25
DATE OF REPORT : June 4, 1973

I. Menthol Transfer

A. Encapsulated Menthol¹

The 3M's encapsulated menthol was sprayed on cigaret filler with and without gum acacia to serve as a binder. Both fillers lost 43% of the menthol during the application and lost an additional 20-30% during cigaret making.

Paraffin oil and triacetin were found to dissolve menthol in sufficient quantity to serve as a replacement solvent for diethylphthalate. However, 3M has prepared a sample of encapsulated menthol without solvent at our request.

B. Menthol Deliveries²

Marlboro Green rods were attached to CA filters containing 0, 6, and 10% triacetin. Menthol deliveries from these cigarettes were determined over a period of fifteen weeks. Two different batches of cigarettes were prepared. In both runs cigarettes with 6% triacetin delivered about 10% more menthol than either 0 or 10% triacetin on the filters. This effect is being investigated further by having additional samples made with smaller increment of triacetin to verify the above observation.

II. AFX³

The hexane extract of Angelica root was separated by column chromatography on silicic acid and the fraction containing the odor component was further purified by gas chromatography and the sample submitted to Analytical Division for identification. The compound was identified as lignestilide and its saturated and aromatic analogues.

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III. Carbon Taste⁴

The descriptive panel re-evaluated the addition of H₂S and acetaldehyde to carbon filtered cigarettes. The panelists found no difference between control cigaret and the control cigaret plus acetaldehyde or H₂S.

IV. References

- | | |
|-----------------|------------|
| 1. 6260 | 69, 81, 88 |
| 2. 6260 | 76, 77, 79 |
| 3. 6193
6271 | 7, 9
7 |
| 4. 6260 | 70-73 |

R. M. Ikeda /ec

R. M. Ikeda

/ec

2022158667

CHARGE NUMBER: 2500
PROGRAM TITLE: SYNTHESIS OF TOBACCO ADDITIVES
PERIOD COVERED: May 1 - May 31, 1973
PROJECT LEADER: S. C. Clough
DATE OF REPORT: June 4, 1973

I. FLAVOR RELEASE PROGRAM

The custom synthesis and purification of ethyl α,α -dimethyl- β -hydroxy- β -(4-methoxyphenyl)propionate (CR-1214) have been completed, and the material has been delivered to Flavor Development.¹ Thermal degradation of CR-1214 has been shown to occur at 270-275°C (DTA, DSC) under static conditions.² The thermal degradation products, the feasibility of scaleup procedures, and the definition of the purity specifications for this cherry flavor release compound are currently being investigated.³

II. NICOTINOID CHEMISTRY

The synthesis of 2',3',3',4',4',5'-hexadeutero-5'-carbomethoxynicotine was completed for a study which shows the involvement of a pyridyl proton in the mass spectral fragmentation pattern. This completed current efforts towards a paper to be submitted to "Organic Mass Spectrometry."⁴

Two new compounds, 5'-carbomethoxynornicotine (CR-1298) and 5'-carboxamidonornicotine (CR-1299) were prepared for use as potential intermediates in the synthesis of 5'-cyanonicotine.⁴

N-cyclopropylnicotinaldehydeimine was prepared as a potential precursor of 2-(3-pyridyl)-3,4-dihydro-2H-pyrrole.⁵ If the latter is stable, it would serve as an intermediate to 5'-substituted nicotines; if the double bond spontaneously shifts into conjugation, this synthesis represents a one-step route to myosmine.

Improvements were made in the procedure for the methylation of 2-(3-pyridyl)azetidines and a larger quantity of the methyl analog was prepared.⁶ The d,l-pyridylazetidines (methyl and nor), d,l-nicotine, and d,l-nornicotine are to be shipped to Woodard Research Corporation for testing purposes.

III. REFERENCES

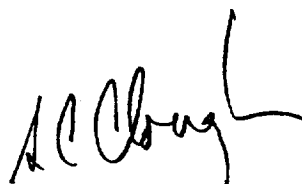
1. Memo from E. G. S. Rundberg, Jr. to T. E. Majewski, May 14, 1973.

2022158668

Page 2.

2. W. Ryan, Notebook 6154.
3. E. Rundberg, Notebooks 6217 and 6327.
4. J. DeBardleben, Notebook 6216.
5. W. Edwards, III, Notebook 6200.
6. H. Secor, Notebook 6195.

/slb



2022158669

CHARGE NUMBER: 2501
PROGRAM TITLE: NUCLEAR AND RADIOCHEMISTRY OF SMOKE
PERIOD COVERED: May 1 - May 31, 1973
PROJECT LEADER: R. W. Jenkins, Jr.
DATE OF REPORT: June 4, 1973

The growth run of $^{14}\text{CO}_2$ tobacco has been successfully completed. The two burley plants have been harvested and are presently being cured. The bottom leaves from the two bright plants have been harvested and the top leaves left to continue their growth in the resealed chamber.

The Waters liquid chromatograph has been placed into operation.

Additional samples of MF blend, labelled with a scintillating phosphor, have been analyzed for Project 1801.

The Finnigan mass spectrometer has been connected to our GC-smoking machine and after debugging, spectra of the gas phase portion of smoke from IRI cigarettes are being interpreted. Runs will be made using a Chrom 101 and a Parapak Q columns. The identification of the peaks eluting from these columns will aid in interpreting the results of mainstream smoke composition from tobacco with added ^{14}C compounds.

The refrigeration unit has been installed in the new small growth chamber.

Preliminary studies of the gross difference between green freeze-dried tobacco and laboratory-cured bright tobacco have been carried out. Moisture determinations on the total samples and TLC studies of the hexane extracts of both samples have been conducted.

REFERENCES

- | | | |
|----|------------|------|
| 1. | R. Bass | 5385 |
| 2. | R. Comes | 5521 |
| 3. | M. Core | 6066 |
| 4. | M. Edmonds | 6250 |
| 5. | B. Francis | 6251 |
| 6. | G. Newell | 5389 |

R. W. Jenkins/slb

/slb

2022158670

CHARGE NUMBER: 4008
PROJECT TITLE: SMOKER SIMULATION STUDIES
PERIOD COVERED: May 1 - May 31, 1973
DATE OF REPORT: June 11, 1973
PROJECT LEADER: George C. Kiritsis

An Annual Report was issued on May 21, 1973.


G. C. Kiritsis

/ gmm

2022158671

CHARGE NUMBER: 4009
PROGRAM TITLE: SMOKE MODIFICATION
PERIOD COVERED: MAY, 1973
PROJECT LEADER: W. A. GEISZLER
WRITTEN BY: A. T. LENDVAY, W. A. GEISZLER
DATE OF REPORT: JUNE 13, 1973

A. FILLER MODIFICATION

Analysis of cigarettes containing carbon-filled TFP added to MF filler shows that TPM, nicotine and puff count decrease with increasing carbon content. Carbon contents of 2 to 11% in filler were tested. The filtration efficiency of the cigarette rod does not change with increasing carbon content, contrary to the behavior of carbon paper in the filler where filtration efficiency increased with carbon paper content. The TPM and nicotine reductions with carbon TFP in the blend can be accounted for by the combined effect of puff count reduction and lower tobacco content of the cigarette. CO delivery does not change significantly with increasing carbon TFP, but HCN delivery appears to rise.¹

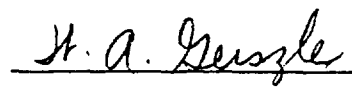
B. WRAPPER MODIFICATION

Sodium and potassium citrate have been compared as burn additives on cigarette paper and found to give no difference in analytical smoking characteristics at the 1% citrate level. A mixture of the citrates performs the same as each citrate alone.² Subjective comparison of the cigarettes is being conducted.

Product development of a cigarette containing CR-1214 to give an improved sidestream aroma has been initiated with a Test Market goal of January, 1974. Cigarettes with the additive on both blend and paper are being compared to determine the preferred method of incorporation. Subjective testing has been initiated to determine the threshold levels of CR-1214 for the smoker and for the observer in a smoke-filled room.

REFERENCES

- 1 - Notebook 6237, p. 67.
- 2 - Notebook 5493, pp. 98-99.


W. A. Geiszler

/gmm

CHARGE NUMBER: 6906
PROGRAM TITLE: BIOLOGICAL EFFECTS OF SMOKE
PERIOD COVERED: May 1 - May 31, 1973
PROJECT LEADER: L. Weissbecker
DATE OF REPORT: May 31, 1973

I. EFFECTS ON TISSUES IN CULTURE

Condensate fractions submitted by members of Project 6908 were assayed for glycolytic activity. Samples of X6D1LP-B and X6D1KX-B were dissolved in water and in acetone. The results are similar, indicating, at least for the B fraction, that there is no interaction with the solvent.¹

	<u>X6D1LP-B</u>		<u>X6D1KX-B</u>	
	<u>H₂O</u>	<u>Acetone</u>	<u>H₂O</u>	<u>Acetone</u>
Glycolytic Activity (% of control)	121.5	109.3	167.1	178.2

II. BENZPYRENE HYDROXYLASE

Lymphocytes obtained from smokers appear to react differently to acetone and BaP than cells from nonsmokers. These observations are based on a limited number of samples.²

III. YEAST CELLS

Optical density vs. cell number has been established for non-synchronous yeast grown in ethanol media. The least squares relationship is: cell number = $3.33 \cdot 10^6 \times OD_{660}$.

This information will be used to pick cell concentration for experiments.³

IV. REFERENCES

- | | | | |
|----|------------|------|-------|
| 1. | D. Clayton | 6307 | 3-42 |
| 2. | M. Minor | 6901 | 72-88 |
| 3. | J. Jones | 6234 | 67-73 |

/slb

L. Weissbecker

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CHARGE NUMBER: 6908
PROGRAM TITLE: SMOKE CONDENSATE STUDIES
PERIOD COVERED: April 27 - May 28, 1973
PROJECT LEADER: W. F. Kuhn
DATE OF REPORT: May 29, 1973

I. CONDENSATE PREPARATION

The 240 port smoking machine, as modified by personnel of the Engineering Services Division, was installed and tested. Its operation is satisfactory.³

Seven cigarette types were processed for internal chemical and biological analyses.³

The whole smoke condensate (WSC) from three cigarette types was collected and is being processed. These samples will be used for in vivo testing which is scheduled for July 1.³

II. CHEMICAL AND BIOLOGICAL ANALYSES

A. Chemical Analysis

The fractionation of WSC from the Kentucky Reference Cigarette and Production RCB, using the Grimmer procedure, was repeated ⁷. In the initial experiments, the nitromethane solubles (Fraction V) were transferred to a silica gel column and subsequently chromatographed with solvents of increasing polarity. The difficulty encountered in transferring Fraction V from production RCB was overcome by adsorbing the sample onto silica gel and placing this silica gel onto the top of the column. This technique resulted in changes in the mass distribution of the column eluates.

The WSC from bright tobacco cigarettes was fractionated by the Whitehead procedure in order to determine the chemical and biological variability as a function of age and storage.^{2,8,9} It has been postulated that storage of WSC in acetone may explain the observed differences in biological activity between our fractions and those received from England. In conjunction with this observation, the WSC received from England will be fractionated to ascertain if our fractionation methodology

2022158674

may explain the difference in distribution of measured biological activity.²

The preparative scale thin layer chromatographic separation of Fraction IV (Grimmer procedure) was performed. Two isolated zones were extracted with ethyl acetate and analyzed on the GC/MS system. The lower zone yielded 19 major components. The electron impact and chemical ionization mass spectra were obtained for 13 of these components. These mass spectra were compared with the reference file at NIH (data phone computer connection) and Philip Morris. Only 2,2-bis-(chlorophenyl)-1,1 dichloroethane was identified. The upper zone requires additional separation.¹

Levoglucozan was identified by infrared analysis in a water-soluble portion of expanded bright tobacco WSC. This identification was confirmed by mild hydrolysis to form glucose as determined by a thin layer separation - color formation method.⁴

Bio-beads S-X12 and S-X8 (exclusion limits of molecular weight 400 and 1,000, respectively) will be used in an attempt to remove high molecular weight materials from smoke fractions prior to gas chromatographic analysis.⁵

Nicotine N-oxide was prepared by reacting nicotine with 30% hydrogen peroxide. It is speculated that this compound could be formed during the smoking process.⁶

The investigation of nitrosamines was continued. It was postulated that the most productive and quantitative method for the determination of dimethylnitrosamine would involve the following steps: (a) cleavage of the nitrosamine using cuprous chloride/HCl, (b) neutralization of the subsequent products in a closed system, (c) reaction of amines formed with sodium nitrite, (d) extraction with methylene chloride and (e) gas chromatographic detection and quantification.³

III. BIOLOGICAL ANALYSIS

Using the *E. coli* procedure, it is apparent that the biological activity of the WSC decreases as a function of time when stored in Triton, however, the water-soluble fraction and water-insoluble fraction are not affected appreciably within the same time interval.¹⁰

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Page 3.

IV. REFERENCES

1.	Einolf	6197	76-78
2.	Gager	6188	82
3.	Hellams	6124	47-48
4.	Katz	6127	74
5.	Levins	6056	57
6.	Millham	6285	
7.	Pages	6259	54-55
8.	Smith	6096	62
9.	Trusdell	6231	38

H. G. Fuhner

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CHARGE NUMBER : 8205
PROGRAM TITLE : Packaging Development
PERIOD COVERED: April 16-May 15, 1973
WRITTEN BY : W. I. Carr
DATE OF REPORT: June 7, 1973

I. Tobacco Pouches - Peelable Heat Seal Closure¹

Storage tests of Bond Street Smoking Tobacco Pouches have shown that the use of a peelable heat seal closure is more effective than the standard double fold closure in preventing the tobacco from drying out. These current tests confirm those made in 1969 and 1970.

The pouch material containing a peelable heat seal strip was suggested as a solution to a problem with short weights in the market place.

Some additional testing is planned with the shape of the heat sealer bar and also with Field and Stream Pouch material which is surface printed material rather than reverse printed glassine as in the Bond Street Pouch material.

II. Export Cases²

Test of shipping cases made from "H" board and Double Wall board indicate that the "H" board case provides at least as much moisture protection as the Double Wall Case after one month storage under Hot-Wet (90°F., 85% R.H.) and Hot-Dry (110°F., 15% R.H.) storage conditions.

"H" board is a water resistant paper board which retains its rigidity under damp storage conditions. The Double Wall corrugated board absorbs moisture and becomes soft under damp storage conditions causing the cases to buckle when they are stacked one on the other.

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III. Cold Storage Warehousing - Extension of Time Beyond Six Months³

Cases of cigarettes (B&H 100's Regular and Menthol, Marlboro King Size Soft Packs and Marlboro Flip Top Boxes) and being stored in the Richmond Cold Storage Warehouse to determine the effects of cold storage (40°F., 60% R. H.) for periods longer than six months.

At the end of seven months, there are no defects which can be attributed to cold storage.

IV. Cold Storage Warehouse Monitoring

Cold storage warehouse monitoring has begun. Samples picked up at the various warehouses throughout the country will be examined in both the R&D Packaging Laboratory and the Quality Control Laboratory as in the previous Cold Storage Program.

V. References

Book	Pages
1. 6248	52-55, 65
2. 6248	33, 34, 58, 59
3. 6248	66, 67



W. I. Carr

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2022158679